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LITERATURE SURVEY ON THE PREPARATION OF METALS  
FROM ORGANOMETALLIC COMPOUNDS

MINORU TSUTSUI AND DONALD LORENZ

Prepared by

Research Division  
College of Engineering  
New York University

Contract No. AF 19(628)-1685

Project No. 4608

Task No. 460802

SCIENTIFIC REPORT NO. 1

March 1963

Prepared for

Air Force Cambridge Research Laboratories  
Office of Aerospace Research  
United States Air Force  
Bedford, Massachusetts

DEC 1963  
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## I. Introduction

This literature survey covers the reported work on the preparation of metals from organometallic derivatives. The main emphasis has been placed on the transition and rare earth metals with thermal or chemical methods of recovery.

A general survey of the organometallic derivatives which have been prepared for these types of metals has been included only to suggest possible avenues of approach in selection of the type which would offer the most desirable properties.

Complete detailed descriptions of organometallic compounds may be found in the books and review articles which have been included in this report.



## II. Types of Organometallic Compounds

In general there are three types of organometallic compounds:

a) ionic, b) covalent and c) delocalized covalent ( $\pi$ -complex). While the physical and chemical properties of organometallics vary widely with the metal and the attached groups, it is possible generally to classify their properties according to the type of bonding.

a) Ionic organometallic compounds are formed by elements of the periodic table in groups IA, IIA, IIIB and the Lanthanides. These compounds are salt-like in nature with the organic radical having some negative charge. This causes these compounds to be extremely reactive toward water and oxygen. Most compounds are nonvolatile and insoluble in organic solvents. The lanthanides are included because the only reported stable organometallic derivatives (1) were formed from the cyclopentadienyl anion and they were found to be ionic though they could be sublimed.

b) Covalent organometallic compounds are formed by elements in the periodic table of groups IB, IIB, IIIA, IVA and VA. The stability of these compounds varies with the metal, (e.g. alkyls of Cu, Ag, Au decompose at low temperatures while those of Sn, Si are stable to high temperature. Aryl substituted compounds are much more stable than the corresponding alkyl compounds. They are soluble in organic solvents and most liquids are distillable.

c) Delocalized Covalent ( $\pi$ -complex) organometallic compounds are produced from the transition elements by making use of their vacant d-orbitals. In general these are sublimable solids, soluble in organic solvents with varying stability toward oxidation and hydrolysis. Some examples of these types of compounds are ferrocene, dibenzenechromium and the metal carbonyls.

### III. Recovery of Metal from Organometallic Compounds

#### 1. Thermal Decomposition

##### a) Delocalized Covalent Organometallics ( $\pi$ -Complexes)

Organometallic derivatives of the transition elements have been thermally decomposed to give metallic mirrors. Dibenzenechromium has been reported (2,3,4) to decompose at 250-300° either in a vacuum or in an inert atmosphere to give a bright metallic mirror. The patents (3,4) cover the use of this method for preparation of conducting coatings or films on glass, glass cloth and resins. In addition other metals (4) can be prepared by thermal decomposition of compounds with the general formula  $\text{Ar}_2\text{M}^y$ .

Ar is an organic hydrocarbon compound containing  
an isolated benzene ring

M is any transition element

y represents the ionic charge of the chemical entity  
and may be 0, 1, or 2.

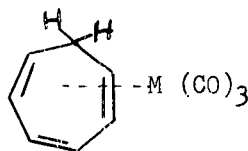
Compounds with the general formula  $\text{ArM}(\text{CO})_3$  M = tungsten, chromium and molybdenum prepared by the reaction of metal carbonyls with aromatic compounds have been reported to produce metallic mirrors (5,6) when heated above 300°.

The preparation of metals from metal carbonyls and metal carbonyl containing compounds often results in contamination from carbon. When nickel is deposited from the carbonyl it contains up to 12% carbon (7) however under some conditions hydrogen gas eliminates this contaminant (8). The effect of

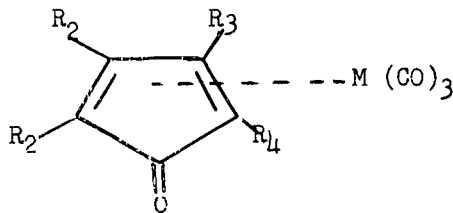
hydrogen and water vapor in reduction of the carbon content in the decomposition of carbonyls of chromium, molybdenum and tungsten have been studied (9, 10, 11).

Carbon contamination has also been observed in the thermal decomposition of cyclopentadienyl metal compounds (12) though this is a reported method for producing metal coatings of manganese, titanium, magnesium, vanadium and nickel (13, 14).

Cycloheptatrienyl metallocarbonyl compounds of chromium, tungsten, manganese, iron and molybdenum are thermally decomposed in an inert atmosphere to give metallic mirrors useful in the production of conducting fabrics and films (15, 16).



Reaction of disubstituted acetylenes with metal carbonyls of iron, manganese and tungsten in neutral nonaqueous media produces compounds of the general formula



M = Fe, Mn and W

Thermal decomposition of these compounds in a non-oxidizing atmosphere produced metallic mirrors of iron, manganese and tungsten (17).

When compounds of the general formula  $[\text{Ar Ar}' \text{Cr(III)}]^+$   $[\text{Co(CO)}_4]^-$  are heated both chromium and cobalt metal are formed (18). A recent patent (19) also describes a method for the production of bimetallic alloys by the decomposition of organometallic compounds. Compounds of the general formula:  $\text{R}_3\text{M}^{\text{iv}} \text{Mn(CO)}_4\text{ER}'_3$  have been decomposed above  $150^\circ$

$\text{M}^{\text{iv}}$  = Si, Ge, Sn, Pb

E = P, Ar, Sb

R = hydrocarbon having up to 18 carbon atoms

R' = a hydrocarbon or oxygen containing hydrocarbon which can be the same or different from R

to produce an alloy of the component metals on a suitable substrate by contacting the heated substrate with the appropriate compound.

#### b) Metals Forming Ionic and Covalent Organometallics

Metallic mirrors of gold, silver and copper are formed in the decomposition of alkyl and aryl organometallic compounds of these elements at low temperatures (20, 21).

Graham and Gatti (22) reviewed the preparation of mercury, lithium, sodium, beryllium, magnesium, boron, aluminum, silicon, germanium, lead,

arsenic, antimony, bismuth, tellurium, zinc and cadmium by the thermal decomposition of organometallic compounds. They have also reported (23) on their attempts to prepare ultrapure samples of magnesium, calcium, mercury, gallium, rhenium and nickel.

Europium metal has been reported to be the product of decomposition of Europium (III) cyclopentadienide when this compound was heated at 150° (24) under vacuum.

## 2. Chemical Method

Nickelocene and Cobaltocene are reduced to the free metal and cyclopentane in cyclohexane solution with hydrogen at 1000 psi in the absence of catalyst. The reduction of nickelocene takes place at 60°, and the reduction of cobaltocene occurs at 160-170° (25).

A study of the hydrogenolysis of organometallic compounds (26) showed that the ease of hydrogenolysis of the metal-carbon bond parallels its degree of polar or ionic character. They also report formation of zinc on hydrogenolysis of diethylzinc and zincacetylide. When a pyridine solution of zinc hydride was heated to 80-100°, zinc and hydrogen were produced.

Hydrogenation of organotin and lead compounds at 60 atmospheres and 200° gives rise to the metal and the hydrocarbon. The tin produced in this reaction was reported to be of high purity (27) but their method of analysis was not very accurate.

When tris(cyclopentadienyl)-dimanganese trinitrosyl was subjected to hydrogenation (28), cyclopentane, ammonia and manganese were found as products.

Lithium aluminum hydride reduction of bis(biphenyl)chromium iodide or benzene-biphenylchromium iodide yields the aromatic hydrocarbon and chromium metal (29).

Acetyl chloride under Friedel Crafts conditions causes the decomposition of ditoluenechromium or dibenzenechromium to give chromium metal and the acetylated aromatic compounds (30). Ferrocene can be cleaved by lithium in ethylenediamine to give iron metal (31). It can also be hydrogenated to cyclopentane, n-pentane and iron at 300° and 280 atmospheres using nickel catalysts (32).

#### IV. Organometallic Compounds of the Transition Elements

Table 1

##### Organometallic Compounds of the Transition Elements

The transition elements form all three types of organometallic compounds (20, 21, 33).

	<u>Sc</u>	<u>Ti</u>	<u>V</u>	<u>Cr</u>	<u>Mn</u>	<u>Fe</u>	<u>Co</u>	<u>Ni</u>	<u>Cu</u>	<u>Zn</u>				
	<u>Y</u>	<u>Zr</u>	<u>Nb</u>	<u>Mo</u>	<u>Tc</u>	<u>Ru</u>	<u>Rh</u>	<u>Pd</u>	<u>Ag</u>	<u>Cd</u>				
	<u>La</u>	<u>Hf</u>	<u>Ta</u>	<u>W</u>	<u>Re</u>	<u>Os</u>	<u>Ir</u>	<u>Pt</u>	<u>Au</u>	<u>Hg</u>				
Lanthinides	<u>Ce</u>	<u>Pr</u>	<u>Nd</u>	<u>Pm</u>	<u>Sm</u>	<u>Eu</u>	<u>Gd</u>	<u>Tb</u>	<u>Dy</u>	<u>Ho</u>	<u>Er</u>	<u>Tm</u>	<u>Yb</u>	<u>Lu</u>
Actinides	<u>Th</u>	<u>Pa</u>	<u>U</u>	<u>Np</u>										

Ionic \_\_\_\_\_

Covalent -----

Delocalized covalent ( $\pi$ -complex) .....

Some general methods of preparation of transition metal organic compounds can be found in the following reviews (20, 22, 34).

A. Alkyl and Aryl Compounds - of the transition elements have only been recently reported (35, 36). It is well known that a number of metal halides react with Grignard reagents, but the resulting derivatives apparently are so reactive that they are difficult to isolate (20, 33, 34). These compounds are sensitive to air and water and the only compounds prepared have been thermally unstable. For this reason, they would be hard to purify and would not be suitable for preparation of ultrapure metals.



Ionic cyclopentadienyl compounds have been prepared from the elements of group IIIB of the periodic table and many of the rare earth elements (37). These compounds though ionic can be sublimed under high vacuum and may thus be purified. It may therefore be possible to prepare ultrapure metal by the decomposition of the pure compounds.

Sigma bonded compounds which are most stable are the ones that are derived from the combination of a metal with cyclopentadiene, carbonyls, nitrosyl or tertiary phosphine groups. Some examples of these types of compounds are:  $\pi$ -(C<sub>5</sub>H<sub>5</sub>) (CO)<sub>3</sub> W-CH<sub>3</sub> ( $\sigma$ ), (Et<sub>3</sub>P)<sub>2</sub>Ni (mesityl)<sub>2</sub>, (CO)<sub>5</sub>Mn(CH<sub>3</sub>) ( $\sigma$ ), (C<sub>5</sub>H<sub>5</sub>)<sub>2</sub>Ti(phenyl)<sub>2</sub> (38, 39, 40).

These compounds show increased stability and possibly could be used for preparation of metals, but they offer no advantages over the  $\pi$  - complex parent compounds.

B. Metal Carbonyls - have been prepared from the following transition elements: V, Cr, Mo, W, Mn, Re, Fe, Ru, Os, Co, Rh, Ir and Ni (20, 41, 42). The preparation of metals from the metal carbonyls offers a possible way to prepare ultrapure metals, if carbon contamination can be minimized.

The preparation of conducting films by thermal decomposition of mixed arene, cyclopentadienyl, cycloheptatrienyl metal carbonyls has already been mentioned. These types of compounds are reasonably stable and can be purified and thus may serve as useful intermediates in the preparation of pure metals from the transition elements.

C. Cyclopentadienyl Compounds - of many elements have been prepared and are summarized in the following table (20, 43, 44).

Table 2

Cyclopentadienyl Compounds

H																	He
<u>Li</u>	Be											B	C	N	O	F	Ne
<u>Na</u>	<u>Mg</u>											Al	Si	P	S	Cl	Ar
<u>K</u>	<u>Ca</u>	<u>Sc</u>	<u>Ti</u>	<u>V</u>	<u>Cr</u>	<u>Mn</u>	<u>Fe</u>	<u>Co</u>	<u>Ni</u>	<u>Cu</u>	<u>Zn</u>	Ga	Ge	As	Se	Br	Kr
<u>Rb</u>	<u>Sr</u>	<u>Y</u>	<u>Zr</u>	<u>Nb</u>	<u>Mo</u>	<u>Tc</u>	<u>Ru</u>	<u>Rh</u>	<u>Pd</u>	Ag	Cd	<u>In</u>	<u>Sn</u>	<u>Sb</u>	Te	I	Xe
<u>Cs</u>	Ba	<u>La</u>	Hf	<u>Ta</u>	<u>W</u>	<u>Re</u>	<u>Os</u>	<u>Ir</u>	Pt	Au	<u>Hg</u>	<u>Tl</u>	<u>Pb</u>	<u>Bi</u>	Po	At	Rn

Lanthanum series

Ce Pr Nd Pm Sm Eu Gd Tb Dy Ho Er Tm Yb Lu

Actinium series

Th Pa U Np Pu Am Crm Bp Cf Es Fm Md No

salt-like .....

$\pi$ -complex \_\_\_\_\_

transitional \_\_\_\_\_

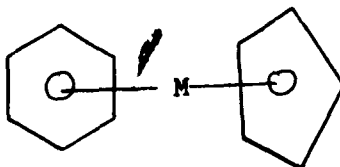
The ionic cyclopentadienyl compounds have been described under alkyl and aryl compounds.  $\pi$ -cyclopentadiene compounds vary as far as stability to heat and air are concerned. A comparison of the  $\pi$ -cyclopentadienes of the first transition shows only ferrocene to be air stable. The titanium compound is thermally unstable decomposing before melting at about 140° and the nickel compound melts at 173° with extensive decomposition.

Thermal decomposition of many different compounds of this type have been reported to give the metal. Hydrogenation of the  $\pi$ -cyclopentadiene compounds might give rise to ultra pure metals.

D. Arene Complexes - of the transition metals have been prepared for the following elements: Cr, V, Mo, W, Fe, Re, Ru, Os, Co, Rh, and Ir (20, 45). These compounds, when the metal is in the zerovalent form are all air sensitive but can be sublimed. The dibenzene metal compounds can be readily converted to the free metal by heating (2, 3, 4). Purification of the cationic arene complexes of the transition elements and then reduction to the zerovalent complex might serve as a useful method of obtaining pure metals. Hafner and Fischer report the separation of iron from chromium (4) by making use of the relative ease of reduction of dibenzenechromium cation over that of the iron derivative.

Many mixed arene-carbonyl metal, and arene-cyclopentadienyl metal compounds have been prepared. Reaction of aromatic systems with metal carbonyls either in an autoclave or under reflux give mixed arene complexes (46, 47). These compounds are more stable to air oxidation than the parent arene metal complexes and for this reason may also serve to produce high purity metals.

Mixed five and six membered aromatic ring metal complexes are relatively stable in air and can be sublimed (48, 49). Decomposition



should give rise to metals but contamination from carbon as is found with cyclopentadiene derivatives may be expected.

E. Olefin Complexes - of the transition metals are confined to the elements in which the  $d$ -shell is filled or nearly filled. These elements include Cu, Ag, Au, Ni, Pd, Pt, Ru and Fe (58).

The olefin complexes of platinum and paladium have been extensively studied because they serve in the preparation of aldehydes and ketones from olefins (50). Reduction of olefin complexes by molecular hydrogen takes place rapidly and quantitatively. Many react with water to give the finely divided metal and carbonyl derivative.

Among the olefins that have been used are cyclohexene, styrene, isobutene, cycloocta-1,5-diene, norbornadiene, dicyclopentadiene, butadiene and many others (20).

$\pi$ -Allyl derivatives of transition metal carbonyls have been prepared for Co, Ni, Pd, Mn and Fe (51, 52). Cycloheptatriene reacts with metal carbonyls to form stable complexes which can be decomposed to metallic mirrors (15, 16, 53). 1,3,5-Cyclooctatriene, just as cycloheptatriene displaces three carbonyl groups from metal carbonyls. However, cyclooctatetraene can only displace two carbonyl groups from molybdenum or iron carbonyl (54, 55, 56, 57).

It may be seen therefore that by formation of the appropriate complexes all the transition elements can be incorporated into organometallic derivatives, theoretically this means that the pure metals of all the transition elements can be prepared by appropriate decomposition of their organometallic complexes.

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AFCLR-63-22 Unclassified report

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1. Chemistry
2. Metal Preparation
- I. Tsutsui, M.
- II. Lorenz, D.H.

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